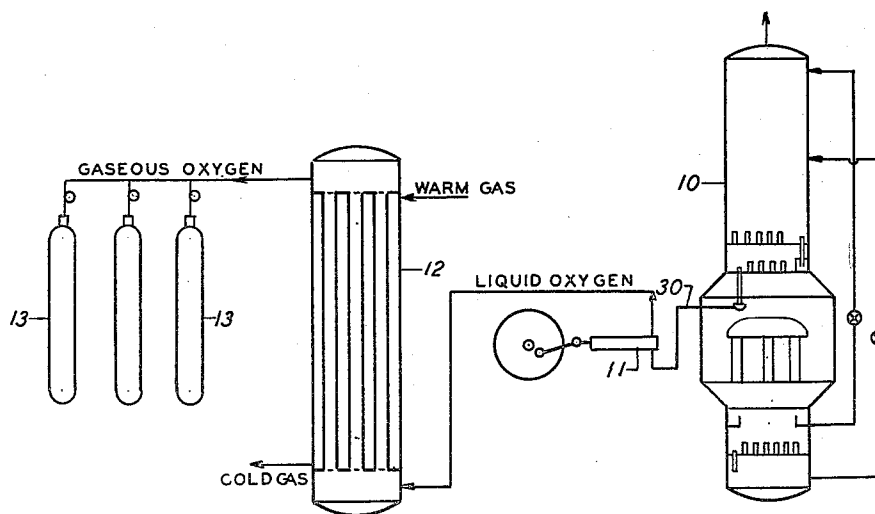
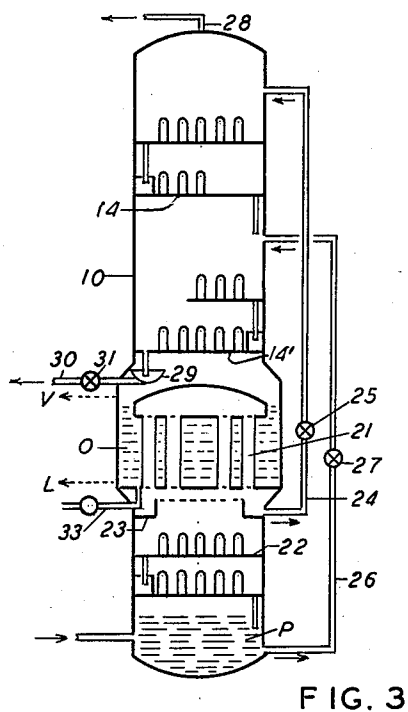
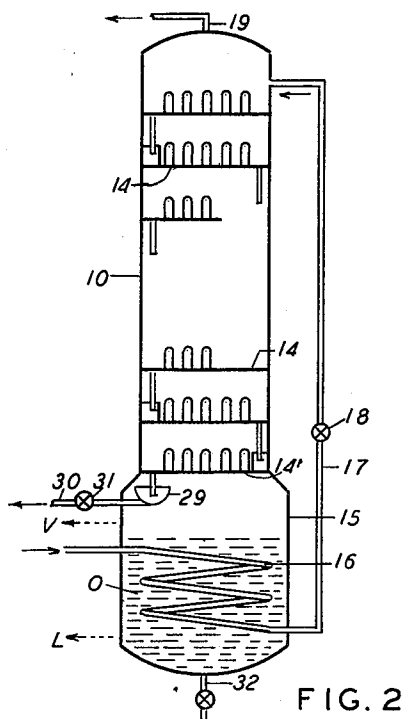


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SEPARATION OF GAS MIXTURES
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SEPARATION OF GAS MIXTURES

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This invention relates to the fractionation of gaseous mixtures, particularly to the fractionation of air containing traces of combustible impurities.

The novel step herein described is most useful and applicable in connection with that type of operation in which oxygen is pumped away from an air fractionating column by a pump adapted to the handling of liquids, is passed in liquid form into a vaporizer in which it is brought to the gaseous state and to atmospheric temperature, and is finally delivered under high pressure into cylinders or other containers for compressed gases.

The objective of the invention is to retain in the fractionating column the largest proportion of the combustible impurities consistent with efficient column operation and thus to retard the accumulation of such impurities in the vaporizing apparatus.

In describing the invention, reference will be made to the appended drawings, in which

Fig. 1 is a flow sheet of an operation such as above described;

Fig. 2 is a highly conventionalized section through a fractionating column of the single-stage type; and

Fig. 3 is a similar representation of a two-stage air fractionating column.

Referring first to Fig. 1, the fractionating column 10 provides (in one manner or another, as will be explained) a stream of liquid oxygen to a piston or plunger pump 11 adapted to handle liquids at high pressure. The pumped stream passes to a vaporizer 12, illustrated by way of example as a tubular interchanger. Air or other warm gas is passed through the shell of the exchanger, around the tubes and in counterflow to the liquid, which is thus gasified and brought up to atmospheric temperature in cooling the air stream. The gaseous oxygen finally passes from the top of the vaporizer to a point of storage of compressed gas, exemplified as a plurality of cylinders 13.

This system is in common use and is highly advantageous as compared with compressing gaseous oxygen by means of gas compressors, the liquid pump and vaporizer being much less costly than a corresponding capacity in gas compression apparatus and requiring only a fraction of the power.

Referring to Fig. 2, the single-stage column consists essentially of a shell 10 provided with a plurality of fractionating plates 14-14, usually of the bubbling type. The lower portion 15 of the

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shell is free from plates but contains a closed pipe coil 16 through which compressed and precooled air is passed by a system of compressors and interchangers not shown. Being liquefied in this coil, the air stream flows through a conduit 17 and expansion valve 18 to the upper end of the column. The bubble plates fractionate the air into gaseous nitrogen, which escapes at 19 from the upper end of the column, and liquid oxygen, which collects in a pool O in the base of the column. The compressed air within the coil supplies heat by which a relatively large quantity of oxygen is vaporized from the pool, this vapor providing the heat by which nitrogen is evaporated out of the descending liquid air and being constantly recondensed and returned to the pool. Columns of this type may be operated over a considerable range of pressures but are ordinarily maintained at about 1.5 atmospheres absolute at the lower end of the column.

In the two-stage column illustrated in Fig. 3 the air supply is introduced into the lower end of the column, which is divided into two sections by a partition including a refluxing condenser 21. In the lower section, which is provided with bubble plates 22-22, nitrogen is evolved and passes up to the condenser, from which it is returned as a liquid. A portion of this liquid is collected on a tray 23 and passes through conduit 24 and expansion valve 25 to reflux the plates 14 in the upper section. The remainder of the liquid nitrogen flows down over the lower plates 22 on which it is continuously reevaporated. The crude oxygen (oxygen-enriched air) collecting in pool P in the base of the lower section passes through conduit 26 and expansion valve 27 into the upper column section at a medial height. In this upper section, substantially pure nitrogen is separated and escapes at 28 while oxygen of the desired degree of purity (controlled by column regulation) collects in a pool O surrounding the condenser 21, which keeps the oxygen constantly boiling. Such columns may be operated over a range of pressures but ordinarily the lower section is maintained at about 5 atmospheres and the upper section at about 1.3 to 1.5 atmospheres absolute.

To this point the apparatus and operations described are conventional and the description merely provides the environment in which the invention later to be described is effective and desirable.

It is generally recognized that the air supply delivered in compressed condition to an air fractionating column may, and usually does, con-

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tain small but appreciable quantities of objectionable impurities. These impurities include water vapor and carbon dioxide which, being incombustible, do not present any problem so far as the avoidance of danger is concerned. They also include carbon monoxide and various hydrocarbons such as methane, ethylene, acetylene, butadiene (biacetylene) and even heavier members of these groups, all of which form highly explosive mixtures with gaseous oxygen when in sufficient concentration. These dangerous substances may occur in minute quantity in the atmospheres of industrial districts and the acetylenes, in particular, are believed to be formed by the cracking of air cylinder lubricating oils.

Of the above substances, carbon monoxide alone has a boiling range below that of oxygen and in greater part passes out of the fractionating column with the gaseous nitrogen. The remainder are liquefied and (with the exception of methane) are solidified in the column and tend to accumulate in the liquid oxygen fraction, in which they are suspended and perhaps to a slight extent dissolved. The following table shows the melting points of these substances, in degrees Kelvin, and also their approximate vapor pressures at 93°, the temperature at which oxygen boils at 1.5 atmospheres, in the pool O of the two-stage column and the single-stage column.

	Vapor Pressure at 93° K.	Melting Point ° K.
Nitrogen.....	4.7 atm. abs.....	66
Carbon monoxide.....	2.5 atm. abs.....	55
Oxygen.....	1.5 atm. abs.....	89
Methane.....	0.2 atm. abs.....	104
Ethylene.....	Below 1 mm. Hg.....	191
Acetylene.....	Below 1 mm. Hg.....	237
Butadiene.....	Below 1 mm. Hg.....	216
Carbon dioxide.....	Below 1 mm. Hg.....	

In the practice of the prior art, oxygen from an air fractionating column has been supplied to a liquid pump in two ways—by withdrawing liquid from below the surface of the liquid pool O, as at L in Figs. 2 and 3, or by withdrawing vapor from above the liquid level, as at V in these figures, and condensing the vapor on its way to the pump. These alternative methods produce quite different results as regards the final disposition of the combustible impurities.

When the liquid is withdrawn as such, the suspended impurities are eliminated from the column at the rate at which they are brought into it with the air feed, and are carried forward with the liquid oxygen into the vaporizer. If the impurities could be depended on to vaporize at the rate at which they are carried into the system, the concentration of combustibles in the gaseous oxygen could never reach a dangerous level and this alternative would be wholly satisfactory. But this continuous vaporization of the high boiling solid substances cannot be depended on, as they tend to accumulate at any point of restricted velocity in the vaporizer. Serious explosions have resulted from the sudden dislodgment of accumulations of such solids, which vaporize almost instantaneously on passage into the warm zone and lead to momentary high concentration.

The alternative of withdrawing a stream of vapor from the column and condensing it on its way to the pump is ideal from the standpoint of retaining the combustibles within the column. Any methane content of the air will vaporize

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when the methane content of the liquid oxygen in the pool becomes somewhat elevated, but the vapor pressures of the more dangerous solid acetylenes is so extremely low at column temperature that they are substantially completely retained in the pool.

The drawback to vapor withdrawal lies in a serious disturbance of normal column operation. Unless a costly external refrigerating system be provided, a column fluid must be used to condense the vapor on its way to the pump. The only fluids having at once a sufficiently low temperature and sufficient heat absorbing capacity for this purpose are the cooled and expanded air feed to a single column (as in conduit 17 above valve 18) or the liquid crude oxygen or crude nitrogen flowing from the high pressure stage to the low pressure stage of a double column (as in conduit 26 above valve 27). The use of either of these coolants for condensing the withdrawn stream of oxygen vapor causes the evaporation of a corresponding quantity of the liquid and deprives the column of that amount of reflux, with a material lowering of fractionating efficiency in any given column.

Neither of these drawbacks can be avoided entirely without incurring the other, but the overall performance of any given oxygen plant may be improved to an important degree in the manner illustrated in Figs. 2 and 3. In this practice the lowermost plate 14' of a single column or of the low pressure section of a double column is caused to drain into an open receptacle 29 from which liquid oxygen flows through pipe 30 to the liquid pump 11 of Fig. 1, this flow being controlled by a valve 31.

As the quantity of liquid flowing from this plate is ordinarily from four to five times the quantity of oxygen contained in the air feed, and therefore of the quantity withdrawn by the pump, from one-fourth to one-fifth of the flow from the plate, and a corresponding proportion of the momentary yield of combustibles, is thus passed to the vaporizer 12, the remainder overflowing into the pool O.

The solids collecting in the pool are harmless so long as maintained at column operating temperature. The quantity of solids so collected is kept within bounds, without interrupting column operation, by withdrawing relatively small amounts of liquid oxygen from the bottom of the pool, as at 32 in Fig. 2 or 33 in Fig. 3.

I claim as my invention:

1. In a liquid air fractionating column having a series of bubble plates each having a downcomer, and a reservoir for the reception of liquid oxygen: a receptacle arranged below the final plate of said series to seal the downcomer from said plate, said receptacle having an opening for the overflow of excess liquid to said reservoir, and a conduit arranged to conduct liquid from said receptacle to the exterior of said column.

2. In a liquid air fractionating column having a series of fractionating plates and a reservoir for the reception of liquid oxygen, the combination comprising downcomer means for the lowermost fractionating plate, a receptacle arranged below the lowermost fractionating plate into which the downcomer means projects, an opening in the receptacle arranged to maintain a liquid level therein to seal the downcomer means and to permit excess liquid to overflow into the reservoir, a conduit arranged to conduct liquid from the receptacle to the exterior of the column, and means controlling the conduit to cause less

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liquid to be conducted from the receptacle than enters the receptacle through the downcomer means.

3. In a liquid air fractionating column having a series of fractionating plates and a reservoir for the reception of liquid oxygen, the combination comprising a receptacle arranged below the lowermost fractionating plate, means for directing a stream of liquid oxygen from the lowermost fractionating plate into said receptacle, means for withdrawing a stream of liquid oxygen from the lowermost portion of the receptacle to the exterior of the column, and control means associated with the last claimed means whereby the

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last claimed stream is less than the first claimed stream and substantially constantly related to the quantity of oxygen separated from the air fed to the column.

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